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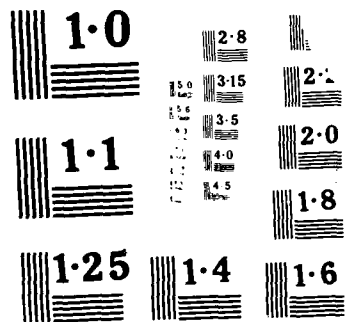
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The wear process in bearings generates a clean active surface. Carbon is known to form readily on catalytic surfaces through the reduction of carbon oxides or hydrocarbon. Carbon, through the adsorption of hydrocarbons, water vapor, or oxygen, becomes an effective lubricant. If these three phenomena can be made to work together, a new concept of high temperature lubrication would be available.

This paper presents laboratory investigations towards the development of this concept. Carbon has been successfully produced through catalytic reduction of ethylene on a variety of metallic and ceramic surfaces containing nickel. This carbon has been shown to reduce friction at a sliding interface at elevated temperatures.

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High Temperature Solid Lubrication by Catalytically Generated Carbon[®]

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The wear process in bearings generates a clean active surface. Carbon is known to form readily on catalytic surfaces through the reduction of carbon oxides or hydrocarbons. Carbon, through the adsorption of hydrocarbons, water vapor, or oxygen, becomes an effective lubricant. If these three phenomena can be made to work together, a new concept of high temperature lubrication would be available.

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INTRODUCTION

Several possibilities exist for high temperature lubrication. They are each listed here with a major drawback which needs to be overcome.

- Synthetic fluids: 500°C maximum use temperature
- Solid lubricants: replenishment
- Molten glasses: Solid at room temperature.

Only solid lubricants show any promise of operating from ambient to above 500°C.

Solid lubricants would have to be replenished in order to provide long life and reliable operation at high temperatures. Various types of replenishment systems have been suggested, and they include such methods as:

1. Suck or Powder Feed
2. Gaseous or Liquid Suspension feed
3. Incorporation in pockets or retainers
4. Gaseous materials which react at the surface.

An example of this last method, which is the subject of

this paper, would be to chemically form the lubricant directly on the bearing surfaces from a gaseous feed material. This concept is not entirely new. Demonstrations have been made using various sulfur or chlorine compounds and oxide films (1), (2) and a U.S. patent has been issued for the gas phase conversion of hydrocarbons to carbon in rolling element bearings at 500°C (3). None of these methods have been used in commercial applications.

This paper deals specifically with the formation and lubricating properties of carbon which is formed by catalytic dehydrogenation or cracking of hydrocarbons on potentially tribological surfaces. In a previous paper (4) we showed that the carbon, formed by catalytic dissociation of ethylene on surfaces containing nickel, would greatly reduce the friction coefficient. This nickel could be in the form of the pure metal, as an alloy constituent, as a metallic transfer film onto a ceramic surface, or as a binder in sintered ceramics. As a result of the deposition of carbon at 500°C the coefficient of friction (μ) in a pin-on-disc friction tester measured under ambient conditions dropped to values as low as 0.08. A high temperature, controlled environment, pin-on-disc friction tester was then constructed which allowed the evaluation of friction and wear at high temperatures simultaneously with the generation of the carbon film. In this apparatus, on surfaces containing nickel, the introduction of ethylene caused a rapid drop in μ down to values as low as 0.08 at 500°C. When the ethylene was replaced by helium, μ slowly returned to its original value. The process could be repeated by reintroduction of the ethylene. Auger surface analysis revealed that the surface contained graphitic carbon, nickel and oxygen.

In this paper, we have used the high temperature apparatus to test a variety of materials and material combinations. These materials included metals, alloys, ceramics and cemented carbides.

BACKGROUND

The formation of carbon on catalytic surfaces is a well known and documented phenomenon. It is sometimes a

problem and sometimes desired and has been studied in relation to such diverse fields as:

1. Deactivation of catalysts in hydrocarbon processing
2. Precipitation of carbon on heat exchanger surfaces of gas cooled nuclear reactors
3. Manufacture of lamp black
4. Coating of waste nuclear fuel pellets
5. Coating of electron tube elements to increase emissivity.
6. Generation of carbon fibers for high strength composites.

Much work has been done in defining the conditions and types of surfaces necessary for the deposition or removal of carbon and on the type of carbon formed. The largest and most carefully documented body of work in these areas is that of hydrocarbon processing catalysis. The majority of the basic research has been done using nickel, iron, or molybdenum surfaces and simple carbon containing gases such as CO, C₂H₄, and CH₄. Examples of this type of work are given in the Refs. (5), (6).

High temperature applications of metals and ceramics require formulations which demonstrate high strength and chemical inertness (or self-protection) in the environment intended for their use. Surfaces are desired which possess minimum porosity and chemical activity. In oxidizing atmospheres, such as exist in gas turbines, materials are generally protected by the addition of aluminum, silicon, chromium, and other trace elements. These materials form oxides which are thermodynamically stable and which are also relatively inert and nonporous. Most commercial ceramics are protected by aluminum or silicon oxides and most metals are protected by aluminum or chromium oxides. Even though these oxides are relatively inert or protective, variations of them are widely used in commercial catalysts and catalyst supports. Additional metals can also be incorporated into these materials by a variety of processes. It may be possible to increase the catalytic activity of the surfaces towards reduction of hydrocarbons and carbon oxides without affecting their high temperature durability. This is really the key of this lubricating process, to develop catalytic activity, while maintaining surface protecting layers.

The continuing regeneration of carbon for lubrication is tied to frictional removal of at least part of the carbon, as thick layers of carbon stop the catalytic reaction. This process is indeed functioning in any rubbing contact. In high temperature combustion engines there is the further complication of an oxidizing atmosphere and contaminants which must be addressed.

Carbon can form on surfaces in several forms depending on the temperature, materials, and kinetics of the system. In catalysis, these different types of carbon have been well documented and have been divided into groups by different researchers (7), (8), (9). These results can be generalized into the following classification for the formation of carbon on nickel from ethylene.

1. Chemisorbed ethylene with increasing dehydrogenation up to 300°C
2. Carbide structure, at 300 to 450°C
3. Graphitic structure, at 350° to 525°C
4. Encapsulating structure (thick graphite layer) at 600 to 1000°C

At high temperatures, carbon solubility increases and surface carbon begins to dissolve into the bulk material. As an example, this occurs at about 650°C for nickel and 1300°C for silicon carbide. Depending on the relative rates of formation and solution, carbon may or may not be present on the surface during this process. On cooling, this carbon can precipitate out of the bulk and form a uniform, glassy graphitic layer (encapsulating carbon) of low surface activity.

Carbon can also be formed by gas phase precipitation at very high temperatures and subsequently settle on adjacent surfaces. This type of carbon can be found even in internal combustion engines (10) but usually is referred to as lamp black.

Of the various types of carbon, graphite is widely used as a boundary lubricant and encapsulating carbon, applied by sputtering, is used as a protective surface coating and lubricant on metallic computer discs. Other types of carbon, such as carbide carbons, are known to be abrasive.

MATERIALS

The purpose of these tests was to explore the behavior of various material combinations in relation to their ability to form lubricating carbon. The tests constituted a variety of combinations of metal and ceramic pins and discs and metallic films applied to the discs. The applied metallic films are designated as "rubbed films" since they were produced by manually rubbing a piece of the metal against a ceramic disc until a visible film was formed. Rubbed films differ from transfer films in that they are produced before a test is begun while transfer films are produced during a test from a metallic pin. Table 1 lists the materials which were evaluated in these tests. The various combinations of these materials actually used are listed in the section on experimental results.

The three gas mixtures used were chosen in such a way as to make one inert toward nickel, and two, carbon forming by containing ethylene, one of which also contained hydrogen. All the atmospheres used contained only slight amounts ($\approx 10^{-4}$ atmospheres) of oxygen because oxygen reduces the catalytic effectiveness of nickel. The atmospheres used are listed in Table 2. The ethylene-hydrogen mixture was chosen because hydrogen is often added in hydrocarbon catalysis to enhance the reaction rate or to maintain catalyst effectiveness. The apparatus was not tightly sealed and gas concentrations were maintained by the flow rates of the various constituents. Argon was used as an inert gas purge for the outer shell and also diffused into the inner chamber. The gas mixtures achieved were analyzed by a residual gas analyzer attached to an ultrahigh vacuum system.

EXPERIMENTAL METHODOLOGY

Our tests were begun with ultrapure multicrystalline nickel for several reasons.

1. It is a single element

TABLE 1—MATERIALS TESTED

CODE	MATERIAL	% NICKEL	COMPOSITION NOTES
A	Nickel Metal	99.997	Ultrapure
B	304 Stainless Steel Metal	10	Commercial Alloy
C	Inconel 600 Metal	77	Commercial Alloy
D	Inconel 718 Metal	52	Commercial Alloy
E	Silicon Nitride Ceramic	—	Multicrystalline, with 8% Yttrium Oxide
F	Aluminum Oxide Ceramic	—	Multicrystalline, 99.5% Pure
G	Aluminum Oxide	—	Synthetic Sapphire
H	Titanium Carbide Cemented Carbide	25	25% Nickel + 7.5% Molybdenum Binder
I	Tungsten Carbide Cemented Carbide	6	6% Nickel Binder
J	Boron Nitride	—	Hexagonal, 98.5% Pure
K	Boron Nitride	—	Hexagonal, 95% Pure
L	Boron Nitride Composite	—	With Titanium Diboride
M	Boron Nitride Composite	—	With 38% Aluminum Oxide

2. Its catalytic behavior is very well documented
3. It is a very effective catalyst
4. It is inexpensive and easy to fabricate.

Nickel by itself is not suitable as a high temperature bearing material because of its low strength at elevated temperatures. However, nickel forms the base for many high temperature alloys and is widely used as a binder in sintered cutting tool materials. Starting with pure nickel created a base line for tests with these materials as well.

Our tests were run with a pin and disc friction tester, (1/8" diameter pin, spherically tipped, 60 gram load, 4 RPM speed, ≈ 2 mm/sec sliding speed.) in a controlled atmosphere at temperatures up to 650°C. In this tester we could simultaneously generate carbon on a surface and evaluate changes in friction brought about by the carbon. Figure 1 shows a schematic diagram of this apparatus and Fig. 2 shows a

typical test run with it. Data output is in the form of strip charts, calibrated to read directly in μ .

In Fig. 2, we see that the test can be divided into several sections. It was begun in an inert atmosphere (gas 1) at room temperature with a μ of about 0.15. Heating to 500°C, brought μ to about 0.55. When an ethylene-containing atmosphere (gas 2) was introduced, μ dropped almost immediately to about 0.10. When this atmosphere was replaced with the inert atmosphere (gas 1), μ increased back up to 0.55 over a 30 minute period. At the end of the test, gas 2 was again introduced and again caused an immediate drop in μ to about 0.10.

EXPERIMENTAL RESULTS

All the tests reported are similar to Fig. 2 and were run at 500°C (except for the temperature effect series) in the atmospheres listed in Table 2. All but one of the tests were begun at room temperature in gas 1 and then heated to the desired operating temperature.

The effects produced were quite large and it is easy to distinguish a successful material combination from an unsuccessful one. Figure 3 shows five typical tests ranging from a strong effect to no effect. All friction records are presented with the initial introductions of ethylene-containing atmosphere (gas 2) aligned since this is where the greatest effect is observed. In some tests this was then followed by gas 3 to see if a further drop in friction could be obtained and all tests ended with gas 1. What follows here are descriptions of various series of tests which demonstrate as-

TABLE 2—GAS MIXTURES USED

GAS NUMBER	DESIGNATION	NOMINAL COMPOSITION
1	Inert	5% Helium 95% Argon
2	Ethylene Containing	50% Ethylene 50% Argon
3	Ethylene + Hydrogen Containing	25% Ethylene 25% Hydrogen 50% Argon

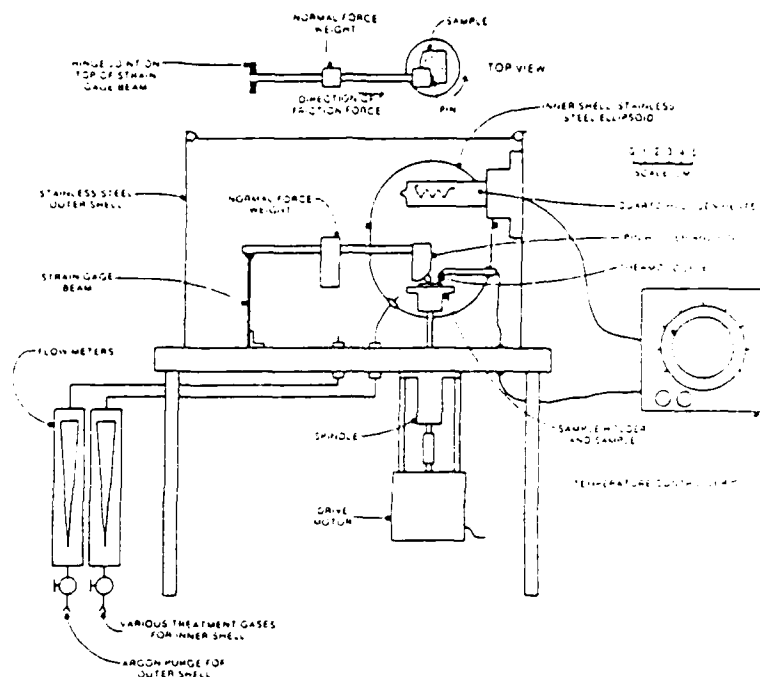


Fig. 1—High temperature pin on disc friction testing apparatus

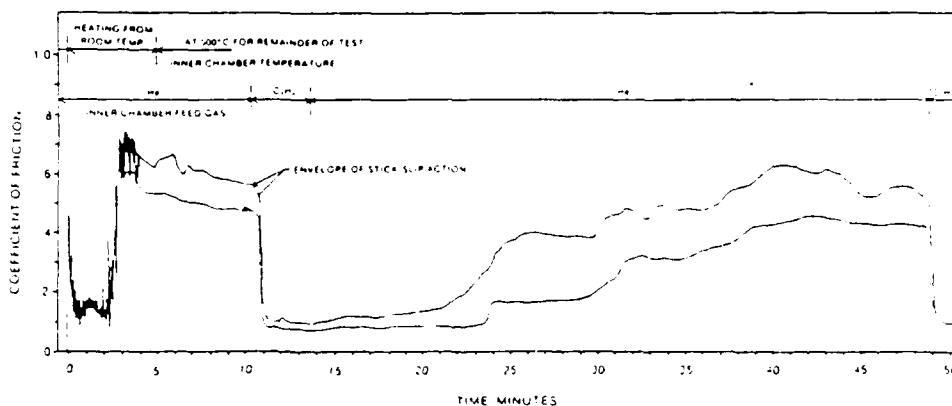


Fig. 2—Typical high temperature pin on disc test

Material Key	Gas Key
pin: A: Nickel	1 Helium + Argon
disc: E: Silicon Nickel	2 Ethylene + Argon

pects of the lubrication process. These test descriptions are then followed by an overall discussion.

Rubbed Metallic Films on Silicon Nitride

Figure 4 shows two tests in which rubbed metallic films were applied to silicon nitride (material E) before the tests were run. Both tests were run with a sapphire pin (material G). The first film was pure nickel (material A) and the second was Inconel 600 (material C). Both films produced a μ of 0.5 to 0.55 in the inert atmosphere and both dropped at about the same rate when the ethylene-containing atmosphere (gas 2) was introduced. The value of μ for the first film dropped to 0.08 and stayed low for the remainder of the test while μ for the second film dropped to about

0.12 and began to increase almost immediately on reintroduction of the inert atmosphere (gas 1).

Nickel-Containing Materials

Figure 5 shows tests which were run on various nickel-containing discs with sapphire pins (material G). Two of the discs are metallic and two are nickel-bonded cemented carbides. The metals, being softer, showed higher μ in the inert atmosphere (gas 1). On introduction of the ethylene-containing atmosphere (gas 2), μ for both the nickel (material A) and the titanium carbide/nickel/molybdenum cemented carbide (material H) began to drop steadily. The Inconel 718 (material D) and tungsten carbide/nickel cemented carbide (material I) showed only small effects until

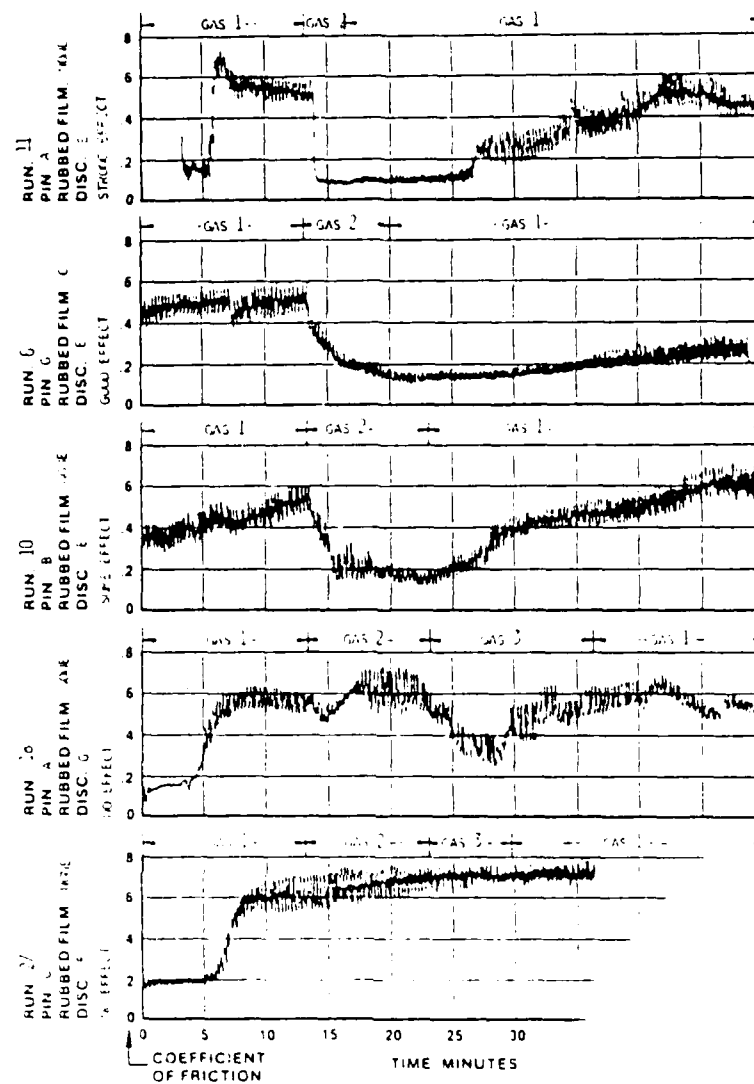


Fig. 3—Range of effects produced

Materials Key

- A Nickel
B 304 Stainless Steel
C Inconel 600

- E Silicon Nitride
F Aluminum Oxide
G Sapphire

Gas Key

- 1 Helium + Argon
2 Ethylene + Argon
3 Ethylene + Hydrogen + Argon

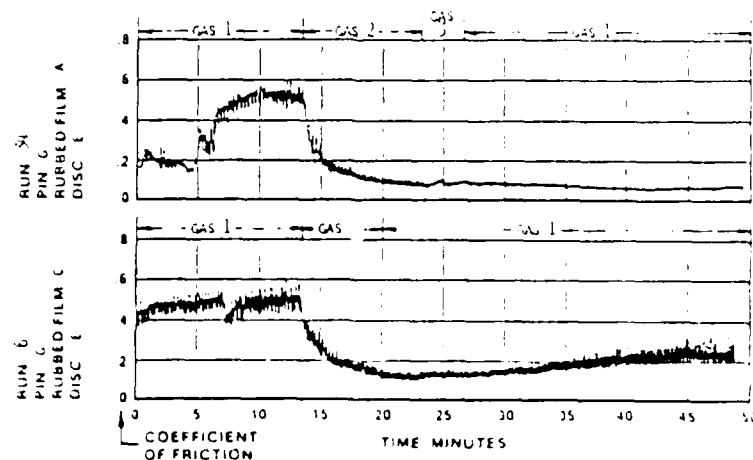


Fig. 4—Comparison of rubbed films on silicon nitride with sapphire pins

Materials Key

- A Nickel
C Inconel 600

- E Silicon Nitride
G Sapphire

Gas Key

- 1 Helium + Argon
2 Ethylene + Argon

- 3 Ethylene + Hydrogen + Argon

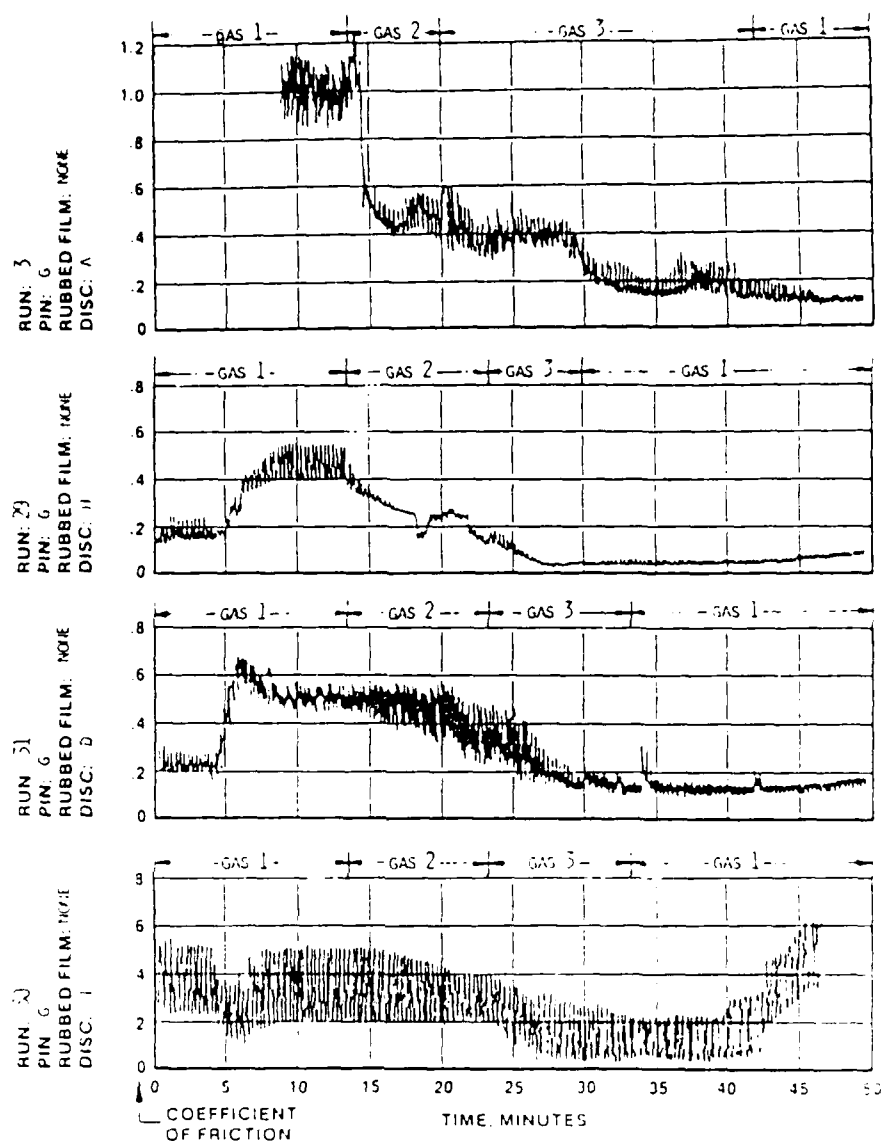


Fig. 5—Discs of nickel containing materials run with sapphire pins

Materials Key

- A Nickel
- D Inconel 718
- G Sapphire
- H Titanium Carbide + Nickel + Molybdenum
- I Tungsten Carbide + Nickel

Gas Key

- 1 Helium + Argon
- 2 Ethylene + Argon
- 3 Ethylene + Hydrogen + Argon

gas 3 (ethylene + hydrogen) was introduced.

Sapphire and Nickel Pins on Sapphire

Figure 6 shows the results for this test series. When a sapphire pin (material G) was run against a sapphire disc (material G), no effect was noted. When a nickel pin (material A) was substituted, again no significant effect was noted. These sapphire discs were optically polished and perhaps a nickel or carbon transfer film could not establish itself. In the third test, the sapphire disc was scratched with a diamond file (200 grit) to roughen its surface. In this case a strong effect was observed, μ dropped rapidly to the ethylene-containing atmosphere (gas 2) and the sample showed comparatively good wear life in the inert atmosphere (gas 1).

Metallic Pins on Ceramic Materials

Figure 7 compares tests of pins of nickel (material A) and 304 stainless steel (material B) on discs of silicon nitride (material E). The value of μ for the nickel pin test dropped faster and to a lower value (0.10 vs. 0.13) and had slightly better wear characteristics than the SS 304 pin (material B). This was true in spite of the fact that the SS 304 pin test was run with the ethylene + hydrogen mixture (gas 3) which generally enhanced the lubricating process.

Boron Nitride Tests

Some tests were run on boron nitride and boron nitride compositions (materials J, K, L, and M). All of these materials were "machineable" and so quite soft. As such, the

sapphire pins (material G) produced some grooving of the discs. The test results are shown in Fig. 8. The process appears to work on the two pure boron nitrides (materials

J and K) and not on the boron nitride mixtures (materials L and M). These boron nitrides were of a hexagonal crystal structure.

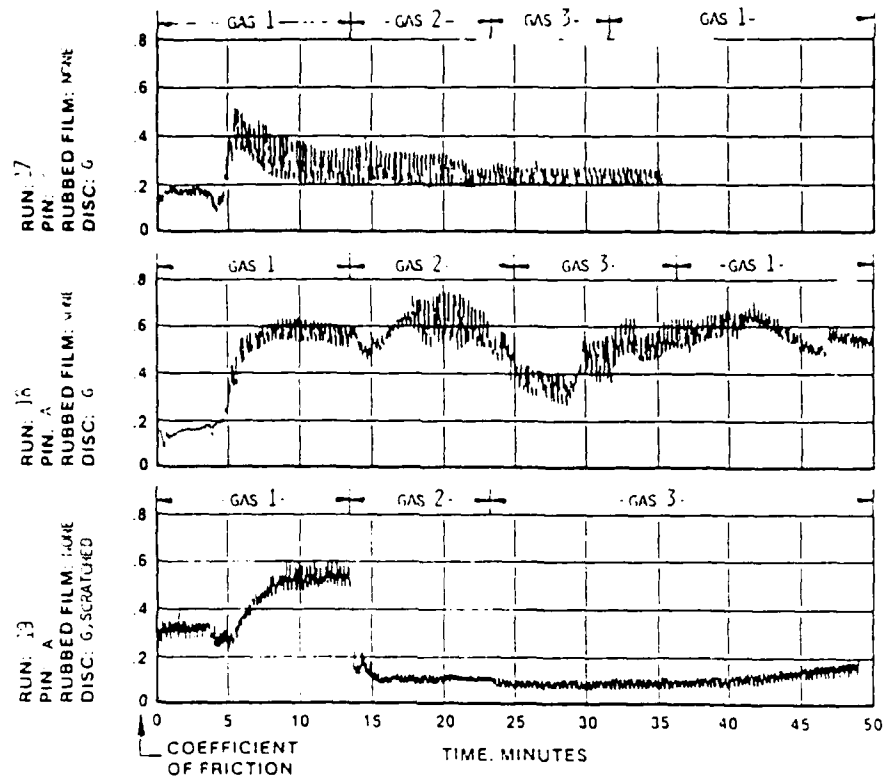


Fig. 6—Sapphire and nickel pins on sapphire

Materials
Keys
A Nickel
G Sapphire

Gas Key
1 Helium + Argon
2 Ethylene + Argon
3 Ethylene + Hydrogen + Argon

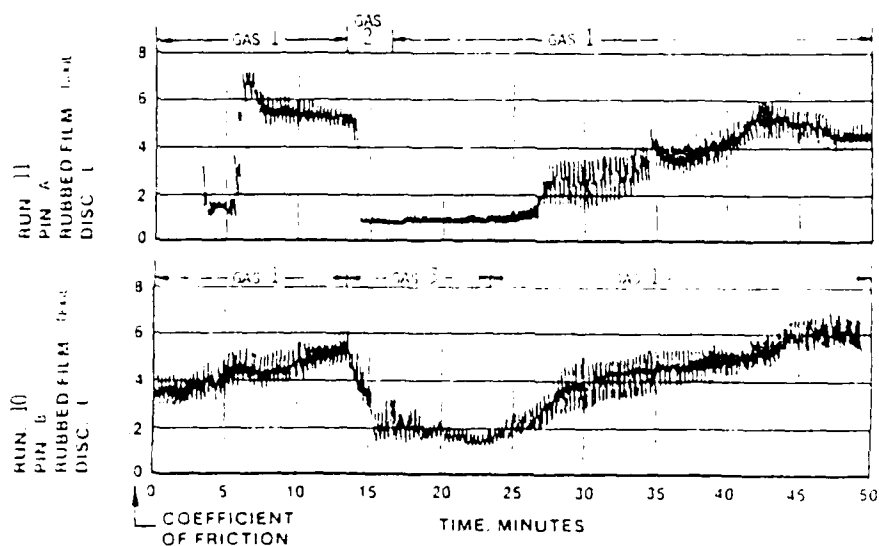


Fig. 7—Metallic pins on silicon nitride

Materials Key
A Nickel
B 304 Stainless Steel
E Silicon Nitride

Gas Key
1 Helium + Argon
2 Ethylene + Argon
3 Ethylene + Hydrogen + Argon

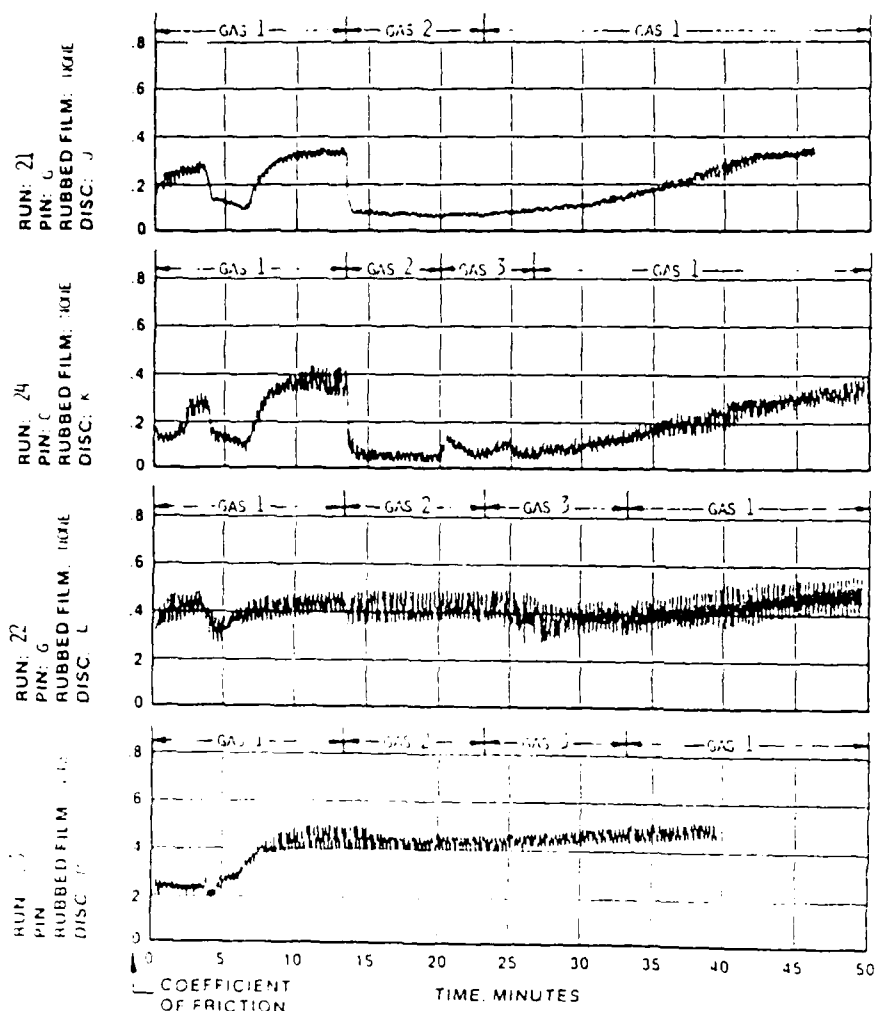


Fig. 8—Boron nitride tests

Materials Key

- G Sapphire
- J Boron Nitride
- K Boron Nitride

- L Boron Nitride + Titanium Dioxide
- M Boron Nitride + Aluminum Oxide

Gas Key

- 1 Helium & Argon
- 2 Ethylene & Argon
- 3 Ethylene + Hydrogen + Argon

Effect of Temperature

Figure 9 shows tests run to evaluate the effect of temperature on the process. These tests were run on silicon nitride discs (material E) with rubbed nickel films (material A) and sapphire pins (material G). Temperatures from 300 to 650°C were run as shown on the strip charts. A summary of the data is given graphically in Fig. 10.

DISCUSSION

The data were evaluated by several parameters which were read directly from the strip charts. A measure of the rate and the extent of the catalytic reaction was gained by how rapidly and how low μ drops when the ethylene-containing atmosphere (gas 2) was introduced and by how smooth the trace became under the influence of the carbon. Some materials such as the Inconels and stainless steels generate protective oxide films which hinder the catalytic pro-

cess. The additional drop in μ achieved when the ethylene + hydrogen atmosphere (gas 3) was introduced gave an indication of the presence of these films. At 500°C, nickel oxide is thermodynamically stable down to an oxygen partial pressure of 10^{-23} atmospheres, chromium oxide is stable down to 10^{-40} atmospheres O_2 , and aluminum oxide is stable down to 10^{-95} atmospheres O_2 (11). The ethylene-containing atmosphere (gas 2) contained oxygen between 10^{-5} and 10^{-2} atmospheres. The ethylene and hydrogen containing atmosphere (gas 3) contained 25 percent hydrogen which, through conversion to H_2O , reduces the effective oxygen partial pressure to about 10^{-40} atmospheres. This means that nickel oxide can be reduced in this atmosphere.

Proper wear measurements were not made. However, some estimate of the wear life or durability of these films was gained by evaluating the time elapsed after reintroduction of the inert atmosphere (gas 1) before μ began to increase.

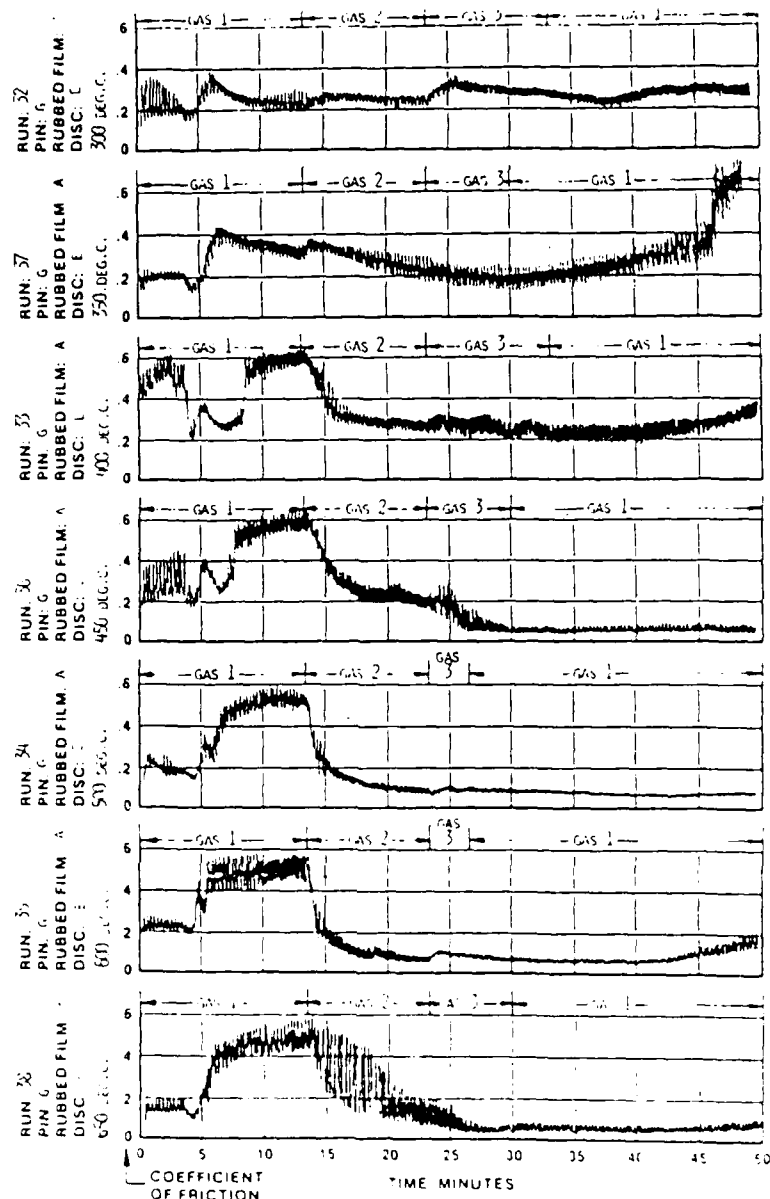


Fig. 9—Effect of temperature

Materials Key

A Nickel
E Silicon Nitride
G Sapphire

Gas Key

1 Helium + Argon
2 Ethylene + Argon
3 Ethylene + Hydrogen + Argon

Pure nickel (material A) appears to be the most effective catalyst for the dissociation of the ethylene-containing atmosphere (gas 2). This is indicated by the rapidity of drop in μ on introduction of this atmosphere as shown in runs 34, 3, 19, and 11 from Figs. 4, 5, 6, and 7 respectively. Materials which contained nickel but in lower concentrations or in conjunction with other oxide-forming materials still showed a drop in μ but not as rapid and to not as low a value. Sometimes the ethylene and hydrogen-containing atmosphere (gas 3) assisted in driving μ down further. Examples of the behavior are given in runs 6, 29, 31, 30 and 10 shown in Figs. 4, 5, and 7 respectively.

Two of the boron nitride tests (runs 21 and 24) show a strong effect on μ with introduction of the ethylene-

containing atmosphere (gas 2) while two runs 22 and 23 showed no effect. The strong effects were observed on runs 21 and 24 in spite of the fact that no visible carbon was formed on the sample surface. These runs are shown in Fig. 8. The boron nitride in these materials is of a hexagonal crystal structure, which has been shown to behave in a similar manner to graphite in sliding contacts (17). It requires an adsorbed material to neutralize the edges of the basal planes so that it can slip easily. Apparently the inert atmosphere (gas 1) did not adsorb while the ethylene-containing atmospheres (gases 2 and 3) did and resulted in lower friction. For the two boron nitride mixtures, perhaps the crystal structure was disturbed by the other constituent so that the boron nitride could no longer act as lubricant.

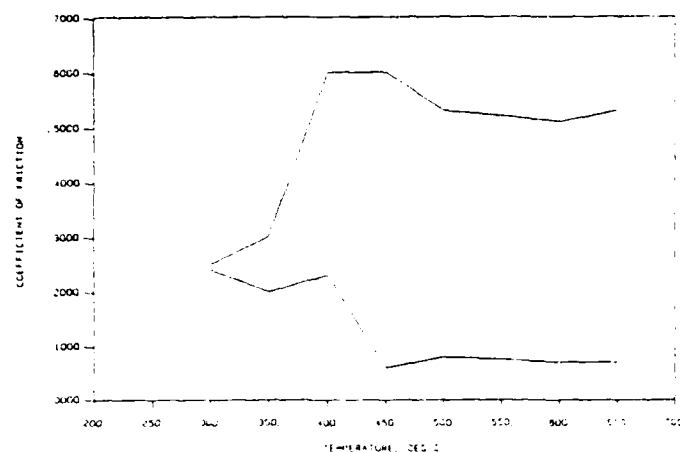


Fig. 10—Summary of temperature test

Material Key

pin G: Sapphire
 rubbed film A: Nickel
 disc E: Silicon Nitride

Gas Key

1 Helium + Argon
 2 Ethylene + Argon
 3 Ethylene + Hydrogen + Argon

TABLE 3—OVERALL SUMMARY OF EXPERIMENTAL DATA

TEST SERIES	PIN	RUBBED FILM	DISC	RUN	TEMP °C	COEFFICIENT OF FRICTION				TIME FOR μ TO 1% MIN
						ROOM TEMP IN GAS 1	HIGH TEMP IN GAS 1	HIGH TEMP IN GAS 2	HIGH TEMP IN GAS 3	
Rubbed Films	G	C	F	6	500	—	0.50	—	0.12	0.7
	G	A	E	34	500	0.18	0.53	0.08	0.08	23.3
Bulk Nickel Mats	G	—	A	3	500	—	1.05	0.18	0.12	11.0
	G	—	H	29	500	0.19	0.46	0.14	0.04	13.3
	G	—	I	30	500	0.36	0.36	0.28	0.15	9.2
	G	—	D	31	500	0.24	0.52	0.40	0.14	10.0
Need for Transfer Films	G	—	G	17	500	0.18	0.24	0.24	—	—
	A	—	G	18	500	0.17	0.56	0.56	0.56	—
	A	—	G*	19	500	0.31	0.55	0.10	—	13.3
Metallic Pins	B	—	E	10	500	—	0.54	—	0.15	0.0
	A	—	E	11	500	0.42	0.53	0.09	—	0.0
Boron Nitrides	G	—	J	21	500	0.28	0.34	0.07	—	0.0
	G	—	L	22	500	0.14	0.14	0.14	0.40	0.0
	G	—	M	23	500	0.23	0.15	0.13	—	0.0
	G	—	K	24	500	0.28	0.36	0.05	—	0.0
Effect of Temp	G	A	E	32	300	0.20	0.25	0.24	0.28	0.0
	G	A	E	37	350	0.24	0.30	0.22	0.20	0.0
	G	A	E	33	400	0.50	0.60	0.26	0.24	0.7
	G	A	E	36	450	0.24	0.60	0.20	0.06	13.3
	G	A	E	34	500	0.18	0.53	0.08	0.08	23.3
	G	A	E	35	600	0.23	0.51	0.07	0.08	13.3
	G	A	E	38	650	0.17	0.53	0.14	0.07	15.0
Bulk Ceramics	G	—	G	17	500	0.18	0.24	0.24	0.24	0.0
	G	—	F	27	500	0.18	0.60	0.60	0.60	0.0

Material Key

A: Nickel
 B: 304 Stainless Steel
 C: Inconel 500
 D: Inconel 718
 E: Silicon Nitride
 F: Aluminum Oxide
 G: Sapphire

H: Titanium Carbide + Nickel + Molybdenum
 I: Tungsten Carbide + Nickel
 J: Boron Nitride
 K: Boron Nitride
 L: Boron Nitride + Titanium Diboride
 M: Boron Nitride + Aluminum Oxide

Gas Key

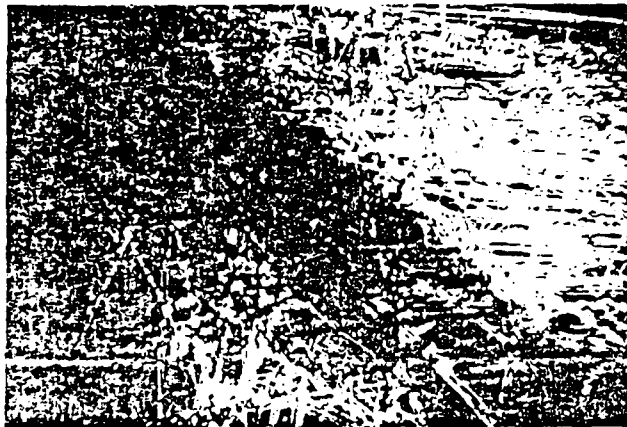
1: Helium + Argon
 2: Ethylene + Argon
 3: Ethylene + Hydrogen + Argon

For nickel, it appears that a temperature of at least 450°C is required to produce μ less than 0.10 with the ethylene-containing atmosphere (gas 2) and that at 650°C, the process may be losing some of its effectiveness as indicated by the slower rate of the decrease of μ on introduction of this atmosphere. In addition, the 500°C test appears to have a better wear life for the carbon film compared to the 600 and 650°C tests.

Table 3 presents an overall summary of the data in tabular form as read from the strip charts and Fig. 11 shows low magnification SEM and optical microscope photographs of representative samples. The appearance of the carbon on the surfaces and in the wear tracks is clearly visible on some of the samples.

CONCLUSIONS

In conclusion we can say lubricating carbon films can be produced by the catalytic dissociation of ethylene-containing atmospheres on nickel containing surfaces. The nickel can be present as a pure element, an alloy component, a metallic rubbed film, a metallic transfer film, or as a binder in ceramic composites. Parallel to the production of this carbon, we found a drop in μ to values below 0.10 measured at elevated temperatures. The process was successful at low oxygen partial pressures which, however, were still thermodynamically capable of producing oxides on the metals involved and oxygen was found to be a constituent of the films by Auger surface analysis.



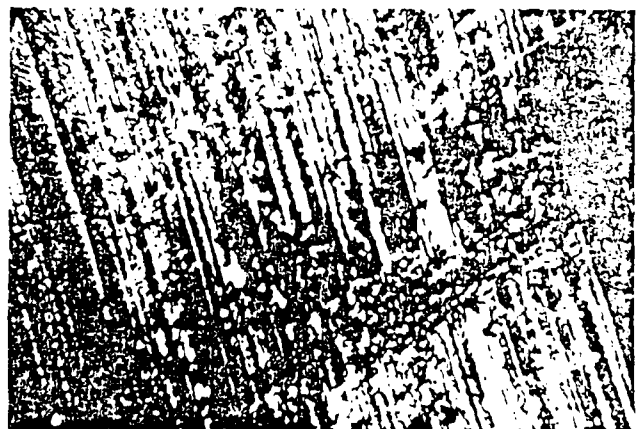
Run 19—Nickel pin on scratched sapphire
Optical—50X
Wear track is upper left to lower right. Carbon has formed on wear track.



Run 24—Sapphire on boron nitride
SEM—100X
Wear track is vertical in center of picture. No carbon formed.



Run 37—Rubbed nickel film on silicon nitride before exposure to ethylene
SEM—100X
No wear track, shows appearance of nickel on silicon nitride surface.



Run 10—304 Stainless steel on silicon nitride
Optical—100X
Wear track is lower left to upper right. Carbon has formed on wear track.

Fig. 11—Typical appearance of wear tracks

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